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THE EFFECT OF PROCESSING CONDITIONS ON THE PHYSICAL PROPERTIES OF SEVERAL POLYURETHANES

by

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ABSTRACT. Experiments were performed, following statistical procedure, to determine the effect of changes in processing conditions on the physical properties of five experimental polyurethanes. Changes in polymer raw materials had more effect on polymer properties than changes in processing parameters, such as curing time, curing temperature, mixing time, and catalyst-addition temperature. Polymer properties, such as gelation time, hardness, and proportional-limit elongation, are more sensitive to changes in processing than other polymer properties, such as tensile strength and polymer-to-metal bond strength. Statistical analysis of variance permitted the effects of two-factor interactions to be estimated.

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FOREWORD

This report describes the effect of processing conditions on the physical properties of five polyurethanes.

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R. J. Landry and J. S. Dinsmore reviewed this report for technical accuracy.

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INTRODUCTION

This report describes experiments performed to determine the effect of processing conditions on the physical properties of five polyurethane polymers now being used at the U. S. Naval Ordnance Test Station as binders, liners, and inhibitors for solid propellants. The polymers, made from reacting a polyhydroxy compound with a diisocyanate, possessed typical polyurethane physical properties.

Since the purpose of the experiment was to evaluate the effect of process variables on polymer physical properties, preparation and testing were conducted under conditions approximating those of commercial manufacture, using appropriate independent variables, such as mixing time and curing time. Data obtained from these experiments have not only helped to define processing conditions that would produce desired polymer properties but also have furnished a basis for making polymers to specific needs.

A statistical design was sought to assure reasonable prediction of variable effects and minimize the number of combinations to be tested.

MATHEMATICAL MODEL

The statistical design that was chosen is expressed by the following mathematical model, which is assumed to best describe the response:

$$X_{ijklm} = \mu + \alpha_i + \beta_j + \gamma_k + (\beta\gamma)_{jk} + \lambda_l + (\beta\lambda)_{jl} + (\gamma\lambda)_{kl}$$
$$+ r_m + (\beta r)_{jm} + (\gamma r)_{km} + (\lambda r)_{lm} + \epsilon_{ijklm} \qquad (1)$$

where

X_{ijklm} = response, the measurement of any dependent variable

μ = the mean of all X_{ijklm} (constant)

α_i = the effect of resin-composition (the difference between resins) fixed variable

β_j = the effect of curing temperature of gelled polymer, fixed variable

γ_k = the effect of temperature of prepolymer at the time of catalyst addition, fixed variable

$(\beta\gamma)_{jk}$ = the effect of interaction between β and γ

λ_l = the effect of mixing time of prepolymer, fixed variable

$(\beta\lambda)_{jl}$ = the effect of interaction between β and λ

$(\gamma\lambda)_{kl}$ = the effect of interaction between γ and λ

r_m = the effect of curing time of gelled polymer, fixed variable

$(\beta r)_{jm}$ = the effect of interaction between β and r

$(\gamma r)_{km}$ = the effect of interaction between γ and r

$(\lambda r)_{lm}$ = the effect of interaction between λ and r

ϵ_{ijklm} = random error that cannot be accounted for (reproducibility of an individual sample)

The values of α_i, β_j, $(\beta\gamma)_{jk}$ $\cdots$, $(\lambda r)_{lm}$ in Eq. 1 are assumed to be negligible.

SAMPLE MODEL

The mathematical model (1) is first estimated by the following sample model:

$$X_{ijklm} = \bar{X} + A_i + B_j + C_k + (BC)_{jk} + D_l + (BD)_{jl} + (CD)_{kl}$$
$$+ E_m + (BE)_{jm} + (CE)_{km} + (DE)_{lm} + e_{ijklm} \tag{2}$$

where

$$\bar{X} \text{ estimates } \mu$$
$$A_i \text{ estimates } \alpha_i$$
$$\cdot$$
$$\cdot$$
$$\cdot$$
$$e_{ijklm} \text{ estimates } \epsilon_{ijklm}$$

and where $\bar{X}$, A_i, B_j $\cdots$ $(DE)_{lm}$ are chosen so that the sum of squares error $(\Sigma_{ijklm} e^2_{ijklm})$ is a minimum.

The sums of squares due to A_i, B_j $\cdots$ $(DE)_{lm}$ are compared with the sums of squares error to determine whether A_i, B_j $\cdots$ $(DE)_{lm}$ are statistically greater than zero.

INDEPENDENT VARIABLES

Data were analyzed using the IBM 7090 computer. The resins were separated into compatible groups before analysis because of unsimilar variances. Independent variables were as follows: 5 replicates of $5 \times 3 \times 2^3$ factorial.

1. A = resin composition

   A_1 = R-6

   A_2 = R-1

   A_3 = R-4

   A_4 = R-9

   A_5 = R-11

2. B = curing temperature, °F

   B_1 = 110

   B_2 = 130

   B_3 = 150

3. C = prepolymer temperature at time of catalyst addition, °F

   C_1 = 140

   C_2 = 160

4. D = prepolymer mixing time, min

   D_1 = 10

   D_2 = 20

5. E = gelled-polymer curing time, hr

   E_1 = 4

   E_2 = 24

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DEPENDENT VARIABLES

The dependent variables measured were

1. Gelation time, min
2. Tensile strength, psi
   a. At maximum stress
   b. At rupture
3. Elongation %
   a. At maximum stress
   b. At rupture
4. Hardness, Shore A
5. Liner-to-metal bond strength, psi
6. Proportional-limit elongation %

EXPERIMENTAL PROCEDURE

Polymer compositions were selected for their range of physical properties. Specifications called for a maximum moisture content of 0.3% (Karl Fischer reagent) and a maximum acid number of 0.50. All reactants were protected from atmospheric moisture. The acid number of all materials was low to prevent excessive CO_2 bubbling. It was also shown that acidic conditions inhibit the gel formations of the reaction with polyglycols and 2,4 toluene diisocyanate (TDI).[1] Mondur TDI was 98.5% of the 2,4 isomer and 2,6 isocyanate analysis, 2.1. Low hydrolyzable chloride content was desirable. The polypropylene glycol (PPG)-2025 had a molecular-weight distribution between 1,800 and 2,100. Castor oil (C-oil), DB grade, was selected because of its low acid number of 1. Other raw materials used were monohydroxyethyltrihydroxypropylethylenediamine (MTDA), phenylbetanapthalamine (PBNA) Neozone D, carbon black (P-33), dipropylene glycol (DPG) molecular weight 134.17, and ferric acetylacetonate (FeAA). A list of the chemicals used and the suppliers are as follows:

Chemical	Vendor
PPG-2025	Carbide and Carbon Chemical Co.
TDI	Mobay Chemical Co.
MTDA	Visco Chemical Co.
PBNA	E. I. du Pont deNemours & Co.
P-33....................	Columbia Carbon Co.
DPG....................	Carbide and Carbon Co.
C-oil....................	Baker Castor Oil Co.
FeAA..................	Aerojet-General Corp.

In general, two methods of resin preparation were employed. R-11 was prepared by one method, and R-1, R-4, R-6, and R-9 were prepared by another. In the preparation of R-11, all components listed in Table 1 were added to the reaction flask, and the mixture was heated and stirred under normal pressure until the temperature reached 230°F. The mixture was then placed under a vacuum (evacuated to below 50 mm of mercury) for 7 minutes and cooled to the desired prepolymer temperature; subsequently, the catalyst was added.

[1]Heiss, H. L., and others. "The Influence of Acids and Bases on the Properties of Urethane Polymers," Industrial and Engineering Chemistry, Vol. 51, 1959, pp. 929–934.

TABLE 1. POLYURETHANE FORMULATIONS

Resin	Composition, parts by weight							
	PPG	DPG	C-oil	TDI	MTDA	FeAA	PBNA	P-33
R-1	....	....	300.0	87.0	....	0.5	....	....
R-4	150.0	....	150.0	56.0	....	.5	....	....
R-6	75.0	25.0	200.0	91.3	....	.5	....	....
R-9	....	80.0	220.0	162.5	....	.5	....	....
R-11	260.0	....	....	30.0	3.0	0.5	1.5	6.0

R-1, R-4, R-6, and R-9 were prepared by simply mixing the reactants at atmospheric pressure without the application of external heating. The exothermic temperature of the reactants provided the necessary temperature for initiating the reaction. After being mixed for a specified time, the reactants were placed under vacuum, brought to the required temperature, and the catalyst added. One portion of the formulation was then poured into a flat mold, and the remainder into a paper cup for gel-time determination.

In the preparation of all polymer compositions, the prepolymer mixing time was defined as the interval between addition of all components to the reaction flask and the degassing operation. Mixing times of 10 and 20 minutes were chosen. The same quantity of resin was prepared in each batch, so that weight would be a constant.

After the prepolymer was prepared in a given evaluation, the catalyst solution of TDI and FeAA was added to promote gelation and subsequent cure. Polymer gelation time is directly dependent on the temperature of the prepolymer at the time of catalyst addition. This variable was introduced into the experiment to evaluate its effect on the physical properties of the polymer. Catalyst-addition temperatures of 140 and 160° F were selected.

Gelled-polymer samples were cured in three constant-temperature electric ovens at 110, 130, and 150°F. To prevent bubble formation at high initial temperatures, the samples were allowed to gel at room temperature (70°F) before being placed in the curing-temperature ovens. No attempt was made to control oven humidity.

The selection of 4- and 24-hour periods as curing times was arbitrary. In pilot-plant production, curing times are based on the process used.

The polymer gelation time was defined as the interval between the instant the catalyst was added to the polymer until the polymer viscosity had exceeded 2 million centipoises, at which time the polymer was nonpourable. The viscosity of the resin during the gelation period was measured using a Brookfield Syncoletric Viscometer, Model RVF. The viscosity measurements were taken in standard laboratory atmosphere, and no attempt was made to control the temperature of the polymer during gelation.

Tensile-strength tests on all polymer samples were performed in accordance with American Society of Testing Materials (ASTM) designation D412-41 for rubbery materials. Test specimens were cut from 8- by 8- by 3/16-inch flat sheets of molded polymer, using a cutting die with a minimum width of 1/4 inch. All samples were stored and tested at 77°F. From the stress—strain data, two values of tensile strength were calculated: the maximum value and the value at rupture. The tensile strength is defined as the load, in pounds, divided by the original minimum cross-sectional area of the sample, in square inches. An average of four measurements was made on each polymer sample.

Hardness measurements on each polymer sample were taken using the ASTM method D-676. When a Shore Durometer, Type A is pressed against a polymer test specimen, the forces exerted by the indented sample and the spring in the instrument are balanced and a value between 0 and 100 is indicated on the durometer scale. Five readings were taken on each sample, and an average value was recorded.

The elastic modulus of a polymer sample is the ratio, within the elastic limit, of stress to the corresponding strain. These data were also obtained as a result of the stress—strain data described in this section. Elastic modulus is reported in pounds per square inch times 10 to the minus 3.

The shear-strength values, polymer-to-steel, were determined by using a modified technique described in ASTM D429-47T. Shear-strength values were obtained on a Dillon tensile-test machine. A crosshead speed of 0.1 in/min was employed.

In the preparation of a bond-strength test sample, the depressed surface on the metal sample holder was filled with liquid resin in the pregel state. Another metal piece was then placed on top of this piece, and the two mating surfaces of each piece were kept parallel by placing the assembly in a vertical press until the polymer gelled.

The proportional-limit elongation is the value of percent elongation of a polymer sample determined as described in the experimental procedure, where the ratio of stress to strain is no longer constant, i.e., where the material ceases to behave as perfectly elastic material. These data were also determined from the stress—strain data.

RESULTS

Experimental runs were evaluated according to a predetermined order corresponding to the statistical design of the experiment. Polymer samples were prepared using conventional laboratory mixers. The possible reaction of the isocyanates with atmospheric moisture during polymer preparation was introduced into the experiment as a random variable. The effect of the laboratory humidity on polymer physical properties was thought to be small since the humidity varied between 10 and 50% during evaluations.

Average values of tensile strength at maximum load and at rupture are as follows:

Formulation	Maximum stress, psi	Stress at rupture, psi
R-11	70.2	70.2
R-4	93.7	93.7
R-6	213.9	213.9
R-1	233.8	233.8
R-9	2,637	2,215

The difference in tensile strength between R-6 and R-1 was insignificant at the 95%-confidence level. In other words, the probability that the difference in values for these two compositions to be as large as it is by chance is greater than 5%. The above table also shows that R-9 was the only composition having a difference in maximum strength at rupture.

Average values of the percent elongation of different polymer compositions are as follows:

Formulation	Elongation at maximum stress, %	Elongation at rupture, %
R-1	62.7	62.7
R-4	41.2	41.2
R-6	99.7	99.7
R-9	24.78	123.06
R-11	334.78	334.35

The difference in percent elongation at maximum stress and at rupture of all resin compositions was significant at the 95%-confidence level. R-9 was the only composition exhibiting a difference in value between percent elongation at maximum and rupture.

Accurate values for modulus of elasticity for the five polyurethanes are as follows:

Formulation	Modulus of elasticity, $psi \times 10^{-3}$
R-1	0.479
R-4	0.282
R-6	0.319
R-9	27.77
R-11	0.030

The difference in elastic modulus between composition R-4 and R-6 was not significant at the 95%-confidence level.

Average values for the gelation time for five formulation studies are as follows:

Formulation	Gelation time, sec
R-4	154
R-1	171
R-6	175
R-9	242
R-11	444

The differences among R-1, R-4, and R-6 were not significant at the 95%-confidence level.

Shore A-Durometer-hardness values are as follows:

Formulation	Hardness, Shore A
R-11	17.5
R-4	42.3
R-6	42.6
R-1	52.9
R-9	greater than 100

The average difference in hardness of R-4 and R-6 was not significant. R-1 is significantly different from R-4 and R-6.

Average values of polymer-to-metal shear strength are as follows:

Formulation	Strength, psi
R-11	85.4
R-4	95
R-1	149
R-6	185
R-9	greater than 300

The difference in average strength between R-11 and R-4 was not significant. Specific value for R-9 is not given because the sample could not be ruptured on the Dillon machine.

Average values for the proportional-limit elongation are as follows:

Formulation	Average proportional-limit elongation, %
R-9	8.43
R-4	15.2
R-1	17.0
R-6	20.3
R-11	57.8

Differences in the proportional-limit-elongation average values of R-4, R-1, and R-6 were not significant.

The data presented in Table 2 were not obtained as part of the statistical experiment described previously. These data illustrate how the physical properties of the polymers change with temperatures.

TABLE 2. PROPERTIES OF POLYURETHANE COMPOSITES
AT LOW TEMPERATURES

Formulation	Test temp., °F	Strength, psi		Elongation, %		
		Max.	At rupture	At max. stress	At rupture	Modulus
R-1	−30	4,254	2,659	10.6	18.6	......
R-4	−30	1,590	1,590	41.0	41.0	......
R-6	−30	4,918	2,533	12.5	22.4	......
R-9	−30	8,922	8,922	14.4	14.4	......
R-11	−30	145	145	398	398	......
R-9	0	6,190	7,370	7.5	101.9	101.5
R-4	0	333	333	20.5	20.5	0.19
R-6	0	......	......	......	......	......
R-1	0	752	752	13.9	13.9	5.67
R-11	0	110	......[a]	296	......[a]	0.08

[a] Did not break.

Physical data obtained from the statistical experiment are presented in the Appendix (Table 3–26).

From the statistical analysis of variance, the following effects were found:

1. In most cases the measured variables have significantly different means corresponding to different resin compositions.

2. The curing temperature of gelled polymer affected the following measured variables:

a. The elongation at maximum stress was affected by curing temperature for R-9 resin only.

b. The proportional-limit elongations for R-11 and R-9 were affected by the curing temperature. For R-9 the curing temperature of 110°F resulted in a significantly higher proportional-limit elongation than those for curing temperatures of 130 and 150°F. For R-11, however, as the curing temperature increased, the proportional-limit elongation increased linearly.

c. Variation of curing temperature had a significant linear effect on the Shore A hardness of R-6, R-1, and R-4. As the temperature increased, the hardness increased.

3. Temperature of prepolymer at the time of catalyst addition affected the following measured variables:

a. The prepolymer temperature of 140°F resulted in a significantly longer gelation time for R-6, R-1, R-4, and R-11 than the temperature of 160°F.

b. The hardness of R-6, R-1, and R-4 at a prepolymer temperature of 140°F was significantly greater than at a prepolymer temperature of 160°F.

4. The mixing time did not significantly affect any of the measured variables.

5. The curing time of the gelled polymer affected the following variables:

a. The 4-hour curing time resulted in a significantly higher proportional-limit elongation for R-9 than that of the 24-hour curing time.

b. The 4-hour curing time resulted in a significantly softer material for R-6, R-1, and R-4 than that of the 24-hour curing time.

c. The 4-hour curing time resulted in a significantly lower bond strength for R-6, R-1, and R-4 than that of the 24-hour curing time.

DISCUSSION AND CONCLUSIONS

Present work indicates that polymers prepared from C-oil or DPG exhibit widely different physical properties than R-11, which is prepared from a polyether, PPG.[2] The results show that R-11 is a soft, low-strength polymer with extremely good elongation. This polymer has maximum strength and elongation if cured at any temperature between 110 and 150°F from 4 to 24 hours. At higher curing temperatures, this material becomes more elastic. R-11 has a fairly long gel time, compared to R-1, R-4, R-6, and R-9. The gelation time may be shortened approximately 40% by increasing the temperature at catalyst addition from 140 to 160°F.

R-9 behaves as a rigid plastic material rather than a true elastomer. DPG, when added to polyurethane formulations, increases the toughness and hardness of the resin considerably. Curing temperatures between 110 and 150°F seem to produce maximum elongation for R-9, but a more elastic material is produced if the material is cured at the lower temperature. The strength and hardness, however, do not seem to be affected by the curing temperature or the curing time. The gelation time is fairly constant between 140 and 160°F. This result is somewhat surprising, but may be due to the high viscosity of the resin at catalyst-addition temperatures between 140 and 160°F. This high viscosity probably inhibits the curing or crosslinking reaction by sterically hindering the surface-active catalyst, FeAA. This effect masks the over-all effect of catalyst-addition temperature. Although shear strengths were not measured in this experiment, they were found, in later work, to be extremely good. This compound can be used, therefore, in bonding applications where moderate bond strength is required.

Resin compositions R-1, R-4, and R-6 are somewhat similar in over-all physical properties and are, in general, affected in a similar manner by the independent variables. In general, the hardness of these resins is sensitive to curing temperature, curing time, and temperature of prepolymer at catalyst addition. Also, better shear strengths can be obtained by curing these resins

[2] Plastics Laboratory Technical Report 31-A. "Plasticized Modifications of Dipropylene Glycol, Castor Oil, m, Tolylene Diisocyanate Polyurethanes as Potting Resins," by R. E. Christenson and D. S. Trifon, Princeton, N. J., Princeton Univ. Press, 1958.

at 150°F instead of curing them at lower temperatures. From Table 2, it can be seen that the composition of R-4 is between that of R-1 and R-11. However, this resin has only 65% of the tensile strength and 50% of the elongation of R-1, and 14% of the elongation of R-11. Composition R-1 is used mainly as a potting compound and for transparent-motor-tube liners.

R-11 polymer gave significantly higher elongation at lower temperatures. This unusual property permits the use of this material at low temperatures where a high degree of elongation is required.

The mixing time did not affect the physical properties of the polymer compositions to a significant extent. This indicates that the reactions are nearly complete after 10 minutes mixing time. This means a rigid control of polymer mixing times is not required.

Gelation times, in general, were too short for large-scale processing. Since the catalyst-addition temperature had such a pronounced effect on gelation time, it seems reasonable to achieve longer gel time by lowering the polymer temperature at which the catalyst is added. Since the above effect is assumed to be linear, gelation times at other temperatures may be obtained by extrapolation of the data; gel time also can be increased by decreasing the catalyst concentration.

In general, varying the concentration of PPG, DPG, and C-oil when reacted with an excess of of TDI will produce resins of widely different physical properties. These properties can be altered somewhat by varying the processing conditions. As was expected, changes in resin compositions resulted in greater variation of physical properties than changes in processing conditions.

Appendix
THE ANALYSIS OF VARIANCE

The following code is used with the analyses:

Code	Effect	Inequality
$\pm$	Not significant	$(P < 0.95)$
+	Significant	$(0.95 < P < 0.99)$
++	Highly significant	$(0.99 < P < 0.999)$
+++	Very highly significant	$(P > 0.999)$

The inequalities in parentheses, such as $(P < 0.95)$ and $(0.95 < P < 0.99)$, are probabilities (P). If, for example, when + appears, one would say, "There is an effect," the statement has a 95% probability of being correct. When the work "significant" (Sig.) appears, it denotes statistical significance that may or may not be of engineering significance.

Entries under "Means" for Main Effects are in the order of several levels for each of the source of variations for which they are means. Under "Means" for Two-Factor Interactions, the entries (1,1), (2,2), etc., indicate the respective levels of the two factors identified under Source of variation. Interacting factors and corresponding means are given only for those cases in which statistical significance was found.

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TABLE 3. Maximum Strength and Strength at Rupture, psi, of Resins $A_1 = R_6$, $A_2 = R_1$, and $A_3 = R_4$

Main Effects					
Source of variation	F-ratio	Sig.	Means, psi		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
A	51.86	+++	213.9	233.8	93.7
B_L	0.00	–	....	....	....
B_Q	2.35	–	174.0	183.7	173.8
C	0.20	–	183.2	177.8	......
D	3.77	–	192.3	168.7	......
E	0.03	–	179.4	181.5	......

Significant Two-Factor Interactions								
Source of variation	F-ratio	Sig.	Means, psi					
			(1,1)	(1,2)	(2,1)	(2,2)	(3,1)	(3,2)
$B_L C$	6.04	+	157.0	190.9	199.2	188.2	193.4	154.2
CD	4.38	+	207.7	158.6	176.8	178.7	......	......

Experimental error estimate: $S_e = 51.58$; grand mean: 180.47.

The maximum strengths of resin compositions R_6 and R_1 were significantly (+) higher than that of R_4.

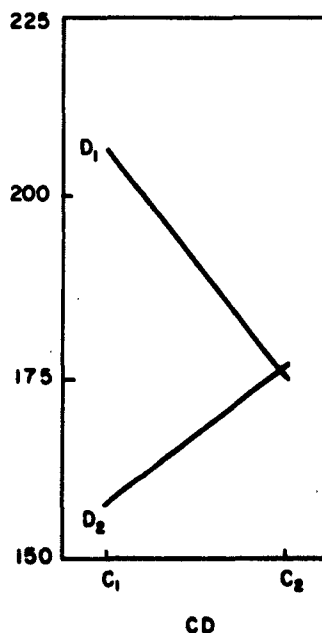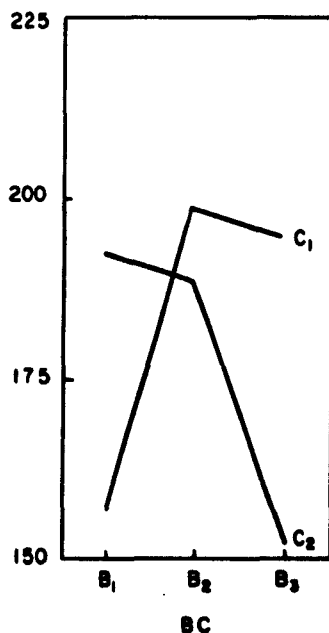The $B_L C$ and CD indicated significance:

TABLE 4. MAXIMUM STRENGTH, PSI, OF RESIN A = R_9

Source of variation	F-ratio	Sig.	Means, psi		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B_L	0.057	—	2,561.6	2,673.1	2,677.5
B_Q	.016	—	....	....	....
C	.151	—	2,560.5	2,714.3	....
D	.184	—	2,552.7	2,722.1	....
E	0.006	—	2,621.7	2,653.1	....

Main Effects

Experimental error estimate: S_e = 968.3; grand mean: 2,637.39.
No significant two-factor interactions and no significant effects were indicated. Independent variables did not alter maximum strength significantly.

TABLE 5. RUPTURE STRENGTH, PSI, OF RESIN A = R_9

Source of interaction	F-ratio	Sig.	Means, psi		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B_L	.74	—	1,981.2	2,376.2	2,289.8
B_Q	.60	—	....	....	....
C	.05	—	2,182.7	2,248.8	....
D	.33	—	2,131.6	2,299.9	....
E	0.16	—	2,156.8	2,274.7	....

Main Effects

Experimental error estimate: S_e = 718.1; grand mean: 2,215.73.
There were no significant two-factor interactions and no significant main effects. The variations of any independent variable did not alter rupture strength significantly.

TABLE 6. MAXIMUM STRENGTH AND STRENGTH
AT RUPTURE, PSI, OF RESIN A = R_{11}

Main Effects					
Source of variation	F-ratio	Sig.	Means, psi		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B_L	0.08	—	62.4	82.5	65.8
B_Q	3.32	—	......	......	......
C	0.76	—	66.1	74.4	......
D	0.17	—	72.2	68.2	......
E	0.64	~	74.0	66.4	......

Significant Two-Factor Interactions								
Source of variation	F-ratio	Sig.	Means, psi					
			(1,1)	(1,2)	(2,1)	(2,2)	(3,1)	(3,2)
$B_Q E$	5.38	+	56.2	68.7	101.9	63.1	64.0	67.5

Experimental error estimate: $S_e = 23.34$; grand mean: 70.23.

The $B_Q E$ interaction indicated significance. Since there were no significant main effects, this interaction is probably not real but merely represents random error:
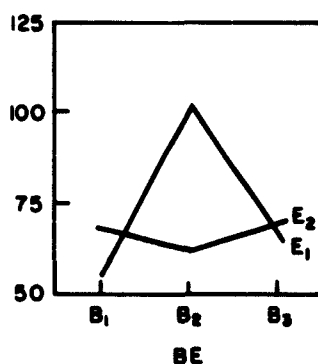
TABLE 7. ELONGATION AT MAXIMUM FORCE AND ELONGATION AT
RUPTURE, %, OF RESINS $A_1 = R_6$, $A_2 = R_1$, AND $A_3 = R_4$

Main Effects					
Source of variation	F-ratio	Sig.	Means, %		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
A	76.05	+++	99.7	62.7	41.2
B_L	2.72	–	64.3	67.1	72.2
B_Q	0.09	–	......	......	......
C	0.59	–	69.4	66.4	......
D	0.66	–	69.4	66.3	......
E	0.92	–	66.0	69.7	......

Significant Two-Factor Interactions										
Source of variation	F-ratio	Sig.	Means, %								
			(1,1)	(1,2)	(1,3)	(2,1)	(2,2)	(2,3)	(3,1)	(3,2)	(3,3)
AB_L	3.37	+	92	93	113	54	68	66	46	40	38
AE	3.39	+	93	106	......	58	67	....	47	36	....

Experimental error estimate: $S_e = 16.6$; grand mean: 67.86.

The mean elongation at maximum force % and the mean elongation at rupture % are significantly (+) different for resins R_6, R_1, and R_4. The means in order from smallest to the largest were R_4, R_1, R_6.

The AB_L interaction significance occurred because R_6 and R_1 change similarly over the levels of B, while resin R_4 has different direction over the levels of B. AE interaction significance resulted because resins R_6 and R_1 change similarly over the levels of E, while resin R_4 has different direction over the levels of E.
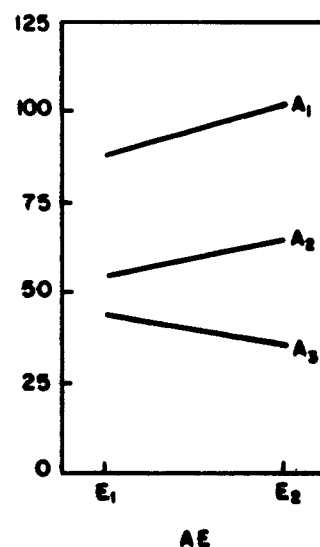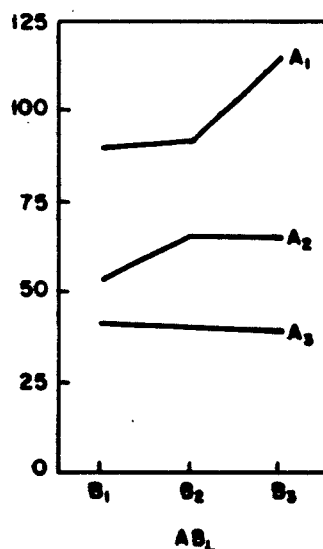
TABLE 8. ELONGATION AT MAXIMUM, %, OF RESIN A - R_9

Main Effects

Source of variation	F-ratio	Sig.	Means, %		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B_L	0.44	—	18.7	45.9	9.7
B_Q	7.23	+	......	......	.....
C	0.08	—	26.3	32.2	.....
D	3.32	—	14.6	34.9	.....
E	1.56	—	17.8	31.7	.....

Significant Two-Factor Interactions

Source of variation	F-ratio	Sig.	Means, %					
			(1,1)	(1,2)	(2,1)	(2,2)	(3,1)	(3,2)
$B_Q D$	7.85	+	24	13	14	78	58	14
$B_Q E$	8.52	+	22	15	16	76	15	44
DE	6.50	+	22	74	14	56	..	..

Experimental error estimate: $S_e = 27.25$; grand mean: 24.78.

The elongation at maximum % was quadratically affected by the curing temperature of gelled polymer for the R_9 resin. The means could not be significantly separated because of insufficient evidence. The arrangement of the means from smallest to largest was $\bar{B}_3$, $\bar{B}_1$, and $\bar{B}_2$.

The $B_Q D$, $B_Q E$, and DE interactions indicated significance. The $B_Q D$ interaction significance is due to the fact that the D_2 level over the B range is quadratically similar to the B_Q main effect while the D_1 level over the B range is quadratically inverted. The $B_Q E$ interaction has similar relations as does the $B_Q D$ interaction.
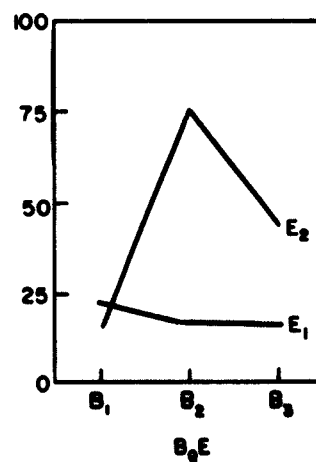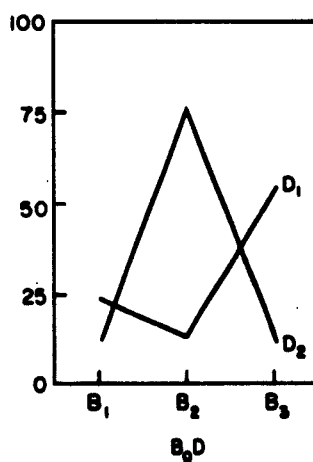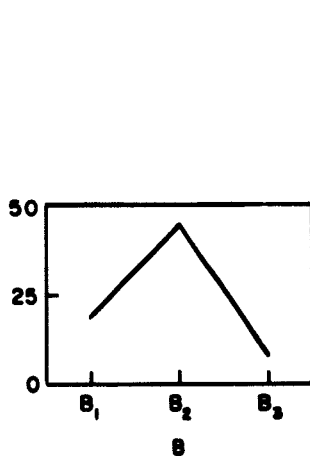
TABLE 9. ELONGATION AT MAXIMUM FORCE AND ELONGATION
AT RUPTURE, %, OF RESIN A = R_{11}

Main Effects

Source of variation	F-ratio	Sig.	Means, %		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B_L	1.36	–	327.7	335.0	370.4
B_Q	0.19	–	....	....	....
C	0.73	–	331.6	357.1	....
D	0.17	–	338.2	350.5	....
E	0.62	–	356.2	332.5	....

Significant Two-Factor Interactions

Source of variation	F-ratio	Sig.	Means, %					
			(1,1)	(1,2)	(2,1)	(2,2)	(3,1)	(3,2)
$B_L C$	10.87	+	364	291	345	325	286	455

Experimental error estimate: $S_e = 73.3$; grand mean: 334.35.

There were no significant main effects. Variations in the independent variables did not alter elongation significantly.

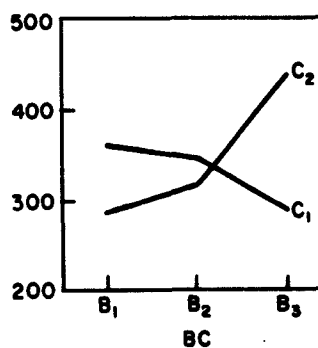The $B_L C$ interaction indicated significance:

TABLE 10. ELONGATION AT RUPTURE, %, OF A = R₉

			Main Effects		
Source of variation	F-ratio	Sig.	Means, %		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B$_L$	0.88	—	109.1	131.4	128.6
B$_Q$	0.49	—	....	....	....
C	3.21	—	138.2	107.9	....
D	1.17	—	132.2	113.9	....
E	3.09	—	138.0	108.2	....

Significant Two-Factor Interactions

Source of variation	F-ratio	Sig.	Means, %					
			(1,1)	(1,2)	(2,1)	(2,2)	(3,1)	(3,2)
B$_Q$D	7.59	+	145.3	73.0	107.6	155.3	143.8	113.4

Experimental error estimate: S_e = 41.5; grand mean: 123.06.

There were no significant main effects. The variations in the independent variables did not alter elongation significantly.

The B$_Q$D interaction indicated significance that is likely to be unreal. However, the points were plotted:
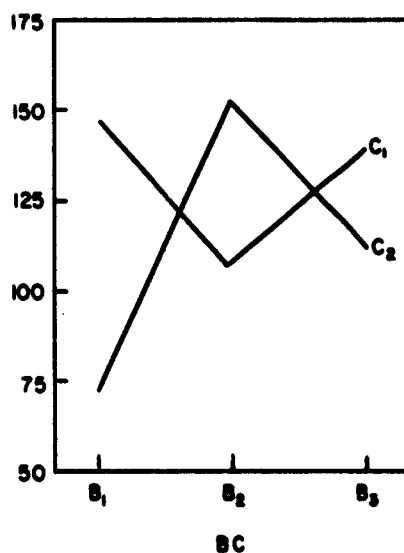
TABLE 11. PROPORTIONAL-LIMIT ELONGATION, %, OF RESINS
$A_1 = R_6$, $A_2 = R_1$, AND $A_3 = R_4$

Main Effects					
Source of variation	F-ratio	Sig.	Mean, %		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
A	1.79	—	20.3	17.0	15.2
B_L	0.92	—	......	......	......
B_Q	0.00	—	16.2	17.5	18.8
C	0.07	—	17.2	17.8	......
D	1.62	—	16.1	18.9	......
E	0.00	—	17.5	17.6	......

Significant Two-Factor Interactions								
Source of variation	F-ratio	Sig.	Means, %					
			(1,1)	(1,2)	(2,1)	(2,2)	(3,1)	(3,2)
$B_L E$	6.03	+	18.8	13.6	18.8	16.2	14.7	22.9

Experimental error estimate: $S_e = 9.48$; grand mean: 17.52.

There were no significant main effects. The variations in the independent variables did not alter proportional-limit elongation significantly.

The indicated significance of $B_L E$ interaction may be unreal since there were no significant main effects. However, the means were plotted:
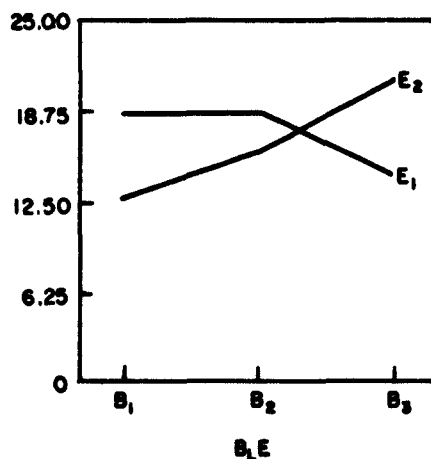
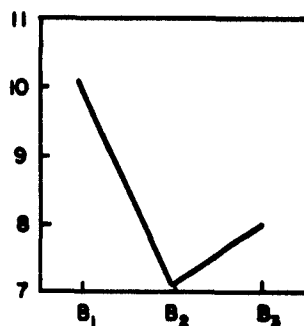TABLE 12. PROPORTIONAL-LIMIT ELONGATION, %,
OF RESIN A = R_9

Main Effects

Source of variation	F-ratio	Sig.	Means, %		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B_L	12.06	+	10.3	7.1	7.9
B_Q	11.63	+	......	....	....
C	0.49	−	8.6	8.2	....
D	2.52	−	8.9	8.0	....
E	35.39	+++	10.1	6.8	....

Significant Two-Factor Interactions

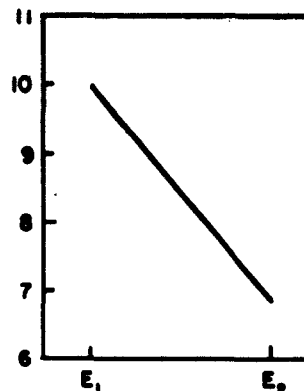| Source of variation	F-ratio	Sig.	Means, %					
			(1,1)	(1,2)	(2,1)	(2,2)	(3,1)	(3,2)
B_LD	6.88	+	11.3	9.2	8.1	6.1	7.2	8.7
B_LE	10.09	+	10.4	10.1	9.6	4.6	10.2	5.7
CD	14.92	++	8.0	9.2	9.7	6.7	....	....
CE	6.50	+	11.0	6.3	9.2	7.3	....	....
DE	12.10	+	11.4	6.3	8.7	7.3	....	....

Experimental error estimate: S_e = 1.34; grand mean: 8.43.

The proportional-limit elongation, %, was significantly affected by variations of curing temperature and curing time of gelled polymer. The curing temperature of 110°F resulted in significantly higher proportional-limit elongation, %, than those curing temperatures of 130 and 150°F (Graph 1). The 4-hour curing time resulted in a significantly higher proportional-limit elongation, %, than did the 24-hour curing time (Graph 2).

The B_LD interaction was due to the similar trend of B_1 and B_2 over D_1 and D_2 while B_3 over D_1 and D_2 had different direction (Graph 3). The B_LE interaction was caused by the similar trend of B_2 and B_3 over E_1 and E_2 while B_1 over E_1 and E_2 had different direction (Graph 4). The CD interaction was caused by C_1 and C_2 having different direction over the range D_1 and D_2 (Graph 5). The CE interaction resulted from C_1 and C_2 having different directions over E_1 and E_2 (Graph 6). The DE interaction was caused by D_1 and D_2 having different directions over the range E_1 and E_2 (Graph 7).
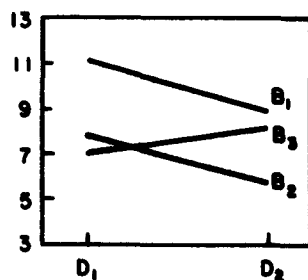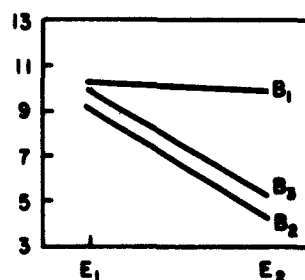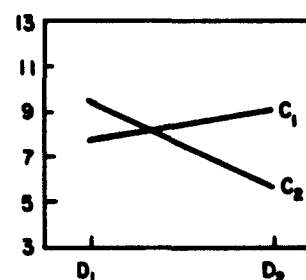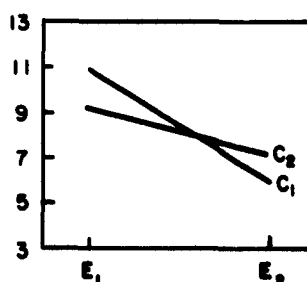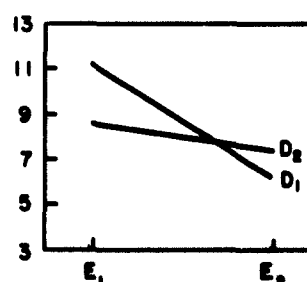
GRAPH 1

GRAPH 2

GRAPH 3

GRAPH 4

GRAPH 5

GRAPH 6

GRAPH 7

TABLE 13. PROPORTIONAL-LIMIT ELONGATION, %,
OF RESIN A = R_{11}

Main Effects					
Source of variation	F-ratio	Sig.	Means, %		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B_L	5.33	+	38.4	51.6	83.3
B_Q	0.30	–	......	......	......
C	0.30	–	62.1	53.5	......
D	0.72	–	51.1	64.5	......
E	0.00	–	58.1	57.5	......

Experimental error estimate: S_e = 38.9; grand mean: 57.80.

The variations of curing temperature of gelled polymer significantly affected the proportional-limit elongation, %. As the curing temperature increased, the proportional-limit elongation increased linearly:
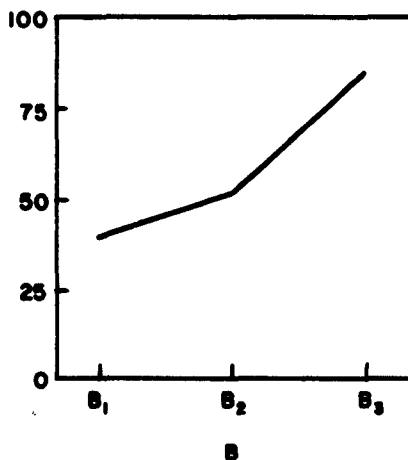
TABLE 14. Proportional Limit, Stress, Psi, of Resins
$A_1 = R_6$, $A_2 = R_1$, and $A_3 = R_4$

Main Effects

Source of variation	F-ratio	Sig.	Means, stress, psi		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
A	14.92	+++	56.7	77.9	38.7
B_L	0.01	−	......	......	......
B_Q	1.81	−	54.7	63.3	55.2
C	0.07	−	57.0	58.5	......
D	0.10	−	56.8	58.7	......
E	0.11	−	58.7	56.8	......

Significant Two-Factor Interactions

Source of variation	F-ratio	Sig.	Means, stress, psi					
			(1,1)	(1,2)	(2,1)	(2,2)	(3,1)	(3,2)
B_LE	5.71	+	65.4	44.0	62.0	64.7	48.8	61.7

Experimental error estimate: $S_d = 24.89$; grand mean: 57.75.

The different resin compositions affected proportional limit, stress, psi, significantly. The order of means from smallest to largest of resins was R_4, R_6, R_1.
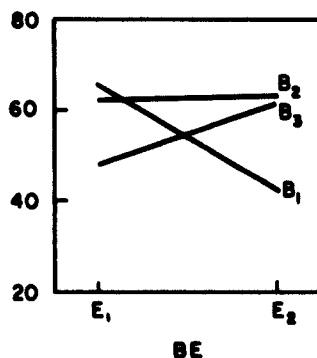
The B_LE interaction is shown:

BE

TABLE 15. PROPORTIONAL LIMIT, STRESS, PSI, OF RESIN A = R_9

Main Effects					
Source of variation	F-ratio	Sig.	Means, psi		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B_L	0.02	–	2,175	1,943	2,118
B_Q	.34	–	....	....	....
C	.43	–	1,969	2,187	....
D	.18	–	2,009	2,148	....
E	0.82	–	2,228	1,928	....

Experimental error estimate: $S_e = 812.4$; grand mean: 2,078.18.
There were no significant two-factor interactions.
The variations of the independent variables did not affect proportional limit significantly.

TABLE 16. MODULUS OF ELASTICITY, PSI × 10^{-3}, OF
RESIN A = R_{11}

Main Effects					
Source of variation	F-ratio	Sig.	Means, psi × 10^{-3}		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B_L	0.56	–	....	....	....
B_Q	1.82	–	0.041	0.053	0.030
C	0.37	–	.037	.045	....
D	0.66	–	.046	.036	....
E	0.01	–	0.042	0.040	....

Experimental error estimate: $S_e = 0.0299$; grand mean: 0.04094.
The independent variables did not significantly affect the modulus of elasticity.
There were no significant two-factor interactions.

TABLE 17. PROPORTIONAL LIMIT, STRESS, PSI,
OF RESIN A = R_{11}

Main Effects					
Source of variation	F-ratio	Sig.	Means, psi		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B_L	5.96	+	13.7	18.6	20.5
B_Q	0.38	−	......	......	......
C	2.43	~	19.4	15.9	......
D	0.01	−	17.5	17.7	......
E	0.06	−	17.9	17.3	......

Significant Two-Factor Interactions								
Source of variation	F-ratio	Sig.	Means, psi					
			(1,1)	(1,2)	(2,1)	(2,2)	(3,1)	(3,2)
$B_Q C$	5.40	+	17.6	9.85	16.7	20.6	23.9	17.2

Experimental error estimate: S_e = 5.56; grand mean: 17.63.

The variations of the curing temperature had a significant linear effect on proportional limit, stress, psi. The order of means from smallest to largest was R_1, R_2, R_3.

The $B_Q C$ interaction was due to the levels of C not having the same quadratic direction over the levels of B:
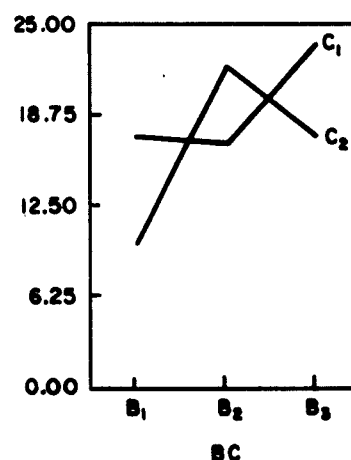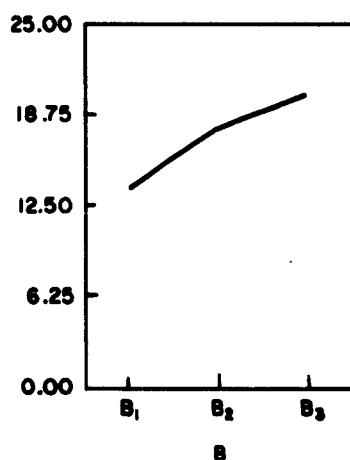
TABLE 18. MODULUS OF ELASTICITY, PSI × 10^{-3}, OF
RESINS $A_1 = R_6$, $A_2 = R_1$, AND $A_3 = R_4$

Main Effects					
Source of variation	F-ratio	Sig.	Means, psi × 10^{-3}		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
A	17.64	+++	0.319	0.479	0.282
B_L	0.70	–	....	....	....
B_Q	2.26	–	.359	.391	0.330
C	0.04	–	.357	.363	....
D	1.51	–	.378	.342	....
E	0.03	–	0.362	0.358	....

Experimental error estimate: $S_e = 0.1224$; grand mean: 0.3599.

The modulus of elasticity was significantly affected by the various resins. Resins R_6 and R_4 resulted in a significantly lower modulus of elasticity than did resin R_1. There were no significant two-factor interactions.

TABLE 19. MODULUS OF ELASTICITY, PSI × 10^{-3},
OF RESIN $A = R_9$

Main Effects					
Source of variation	F-ratio	Sig.	Means, psi × 10^{-3}		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B_L	0.63	–	21.9	33.3	28.1
B_Q	1.53	–	......	......	......
C	0.39	–	25.8	29.7	.....
D	0.63	–	25.3	30.3	......
E	1.04	–	24.5	31.0	.....

Experimental error estimate: $S_e = 15.5$; grand mean: 27.776.

Independent variables did not affect modulus of elasticity significantly. There were no significant two-factor interactions.

TABLE 20. GELATION TIME, SEC, OF RESINS
$A_1 = R_6$, $A_2 = R_1$, AND $A_3 = R_4$

Main Effects

Source of variation	F-ratio	Sig.	Means, sec		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
A	1.32	—	175.1	171.3	154.0
B_L	0.30	—	....	....	....
B_Q	0.01	—	163.3	166.2	170.8
C	33.10	+++	199.2	134.4	....
D	2.07	—	158.7	174.9	....
E	1.72	—	174.2	159.4	....

Significant Two-Factor Interactions

Source of variation	F-ratio	Sig.	Means, sec					
			(1,1)	(1,2)	(2,1)	(2,2)	(3,1)	(3,2)
$B_Q E$	6.00	+	167	159	193	139	162	180
CD	4.83	+	179	220	139	130	......	......

Experimental error estimate: $S_e = 47.78$; grand mean: 166.78.

A prepolymer temperature of 140°F resulted in a significantly higher gelation time than when the prepolymer temperature was 170°F.

The $B_Q E$ interaction is due to the fact that the E_2 and E_1 levels over the range of B are dissimilar.

The CD interaction results from the C_1 and C_2 levels having different directions over the range of D_1:
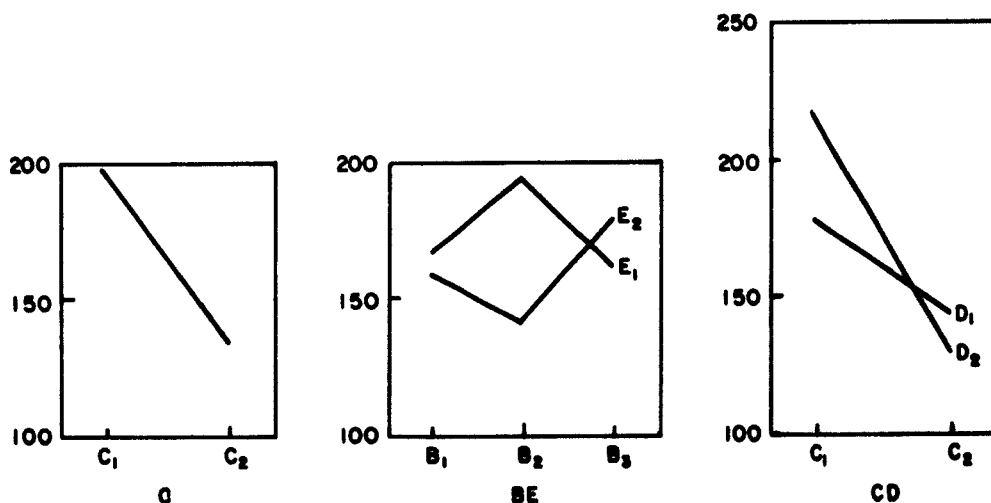
TABLE 21. GELATION TIME, SEC, OF RESIN A - R_{11}

Main Effects					
Source of variation	F-ratio	Sig.	Means, sec		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B_L	1.40	–	433.5	421.5	477.6
B_Q	1.11	–	....	....	....
C	30.60	+++	528.4	360.0	....
D	0.75	–	431.0	457.3	....
E	11.22	+	495.2	393.2	....

Significant Two-Factor Interactions						
Source of variation	F-ratio	Sig.	Means, sec			
			(1,1)	(1,2)	(2,1)	(2,2)
CE	6.45	+	618	439	372	348

Experimental error estimate: $S_e = 74.5$; grand mean: 444.18.

The gelation time was significantly affected by temperature of prepolymer. The lower prepolymer temperature resulted in a longer gelation time than did the higher prepolymer temperature.

A 4-hour curing time resulted in a significantly longer gelation time than did the 24-hour curing time.

The CE interaction significance was the result of C_1 and C_2 having a different direction over the range of E_1:

C

E

CE
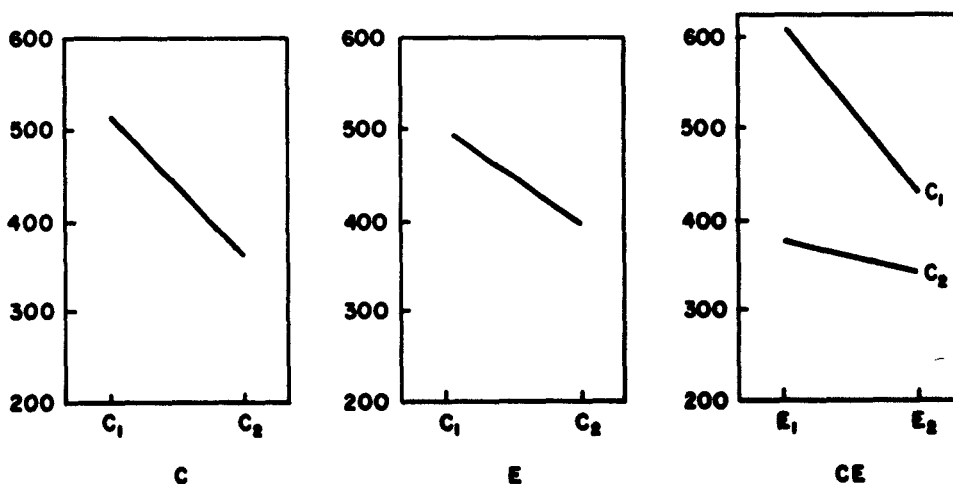
TABLE 22. HARDNESS, SHORE A, OF RESINS
$A_1 = R_6$, $A_2 = R_1$, AND $A_3 = R_4$

Source of variation	F-ratio	Sig.	Means, Shore A		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
A	39.07	+++	52.9	42.6	42.3
B_L	8.81	++	44.1	45.7	48.1
B_Q	0.14	−	......	......	......
C	5.51	+	47.3	44.7	......
D	1.10	−	45.4	46.5	......
E	42.98	+++	42.3	49.6	......

Main Effects heading spans the table.

Experimental error estimate: $S_e = 4.73$; grand mean: 45.95.

There were no significant two-factor interactions.

The hardness (Shore) of R_1 and R_4 was significantly lower than the hardness of resin R_6.

The variations of curing temperature had a significant linear affect on hardness. As the temperature increased, the hardness increased.

The 140°F temperature of prepolymer resulted in a significantly greater hardness than that of the 170°F temperature of prepolymer.

The 4-hour curing time resulted in a significantly softer hardness than did that of the 24-hour curing time. Significant main effects:
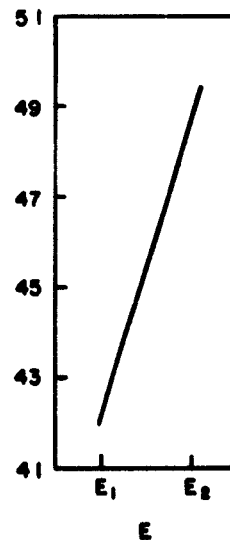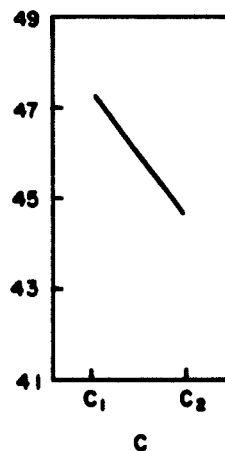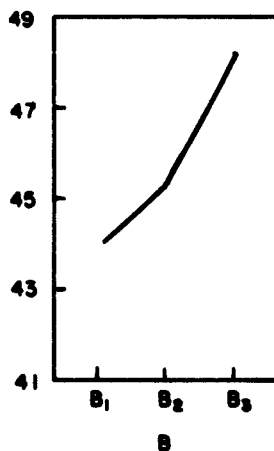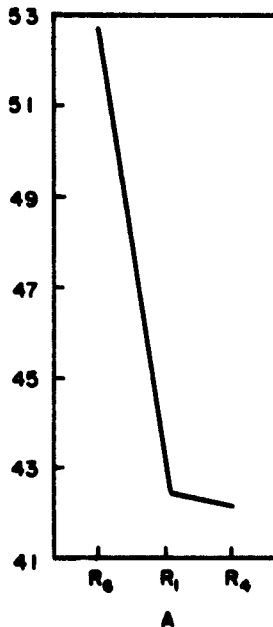
TABLE 23. Gelation Time, Sec, of Resin A = R$_9$

			Main Effects		
Source of variation	F-ratio	Sig.	Means, sec		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B$_L$	0.05	—	153	237	238
B$_Q$	0.02	—	......	......	.....
C	5.06	—	304	181	.....
D	0.73	—	219	266	.....
E	0.11	—	233	252	.....

Experimental error estimate: S_e = 134.5; grand mean: 242.43.
There were no significant two-factor interactions. The gelation time was not significantly affected by variations of the independent variables.

TABLE 24. Hardness, Shore A, of Resin A = R$_{11}$

			Main Effects		
Source of variation	F-ratio	Sig.	Means, Shore A		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B$_L$	1.82	—	16.2	18.1	18.3
B$_Q$	0.39	—	......	......	......
C	1.13	—	18.2	16.9	......
D	0.99	—	16.9	18.1	......
E	2.17	—	16.6	18.4	......

Experimental error estimate: S_e = 2.98; grand mean: 17.51.
There were no significant two-factor interactions. The changes in any of the independent variables had no significant effect on hardness (Shore).

TABLE 25. BOND STRENGTH OF RESINS
$A_1 = R_6$, $A_2 = R_1$, AND $A_3 = R_4$

Source of variation	F-ratio	Sig.	Means, Shore A		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
A	19.93	+++	185.3	149.1	95.0
B_L	0.00	−	147.7	133.5	148.2
B_Q	1.34	−	....	....	....
C	1.34	−	151.0	135.3	....
D	0.51	−	138.9	147.3	....
E	6.35	+	128.3	157.9	....

Main Effects column header spans the table.

Experimental error estimate: $S_e = 49.88$; grand mean: 143.12.

There were no significant two-factor interactions. The bond strengths of the three resins are significantly different in the following order (smallest to the largest): R_4, R_2, and R_6. The 4-hour curing time resulted in a significantly lower bond strength than the 24-hour curing time:
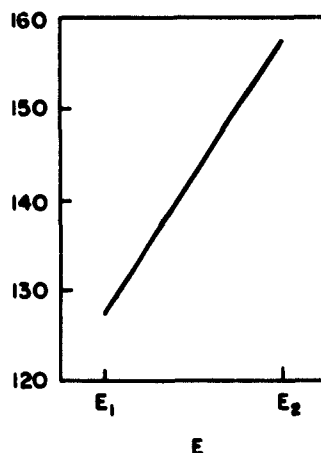
TABLE 26. BOND STRENGTH OF RESIN R_{11}

Main Effects

Source of variation	F-ratio	Sig.	Means, Shore A		
			$\bar{X}_1$	$\bar{X}_2$	$\bar{X}_3$
B_L	1.10	–	84.5	78.2	93.4
B_Q	2.25	–	......	......	......
C	0.16	–	86.8	83.9	......
D	0.13	–	84.2	85.6	......
E	0.10	–	86.4	84.3	......

Significant Two-Factor Interactions

Source of variation	F-ratio	Sig.	Means, Shore A					
			(1,1)	(1,2)	(2,1)	(2,2)	(3,1)	(3,2)
B_Q	6.90	+	74.5	94.8	92.3	64.1	93.8	93.0
B_L	9.17	+	75.0	94.3	75.3	81.0	109.0	77.8
CE	12.66	++	100.0	73.7	72.9	95.0	....	......

Experimental error estimate: S_e = 16.67; grand mean: 85.39.

The variations of the independent variables did not significantly affect bond strength.

The B_QC, B_LE, and CE interactions indicate significance although there were no significant main effects. However, the graphs of the interactions are shown for the judgment of the experimenter.

BC

BE

CE

U. S. Naval Ordnance Test Station
The Effect of Processing Conditions on the Physical Properties of Several Polyurethanes, by Henry T. Sampson and I. S. Kurotori. China Lake, Calif., NOTS, April 1963. 32 pp. (NAVWEPS Report 7934, NOTS TP 2966), UNCLASSIFIED.

ABSTRACT. Experiments were performed, following statistical procedure, to determine the effect of changes in processing conditions on the physical properties of five experimental polyurethanes. Changes in polymer raw materials had more effect on polymer properties than changes in processing parameters, such as curing time, curing tempera-

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ture, mixing time, and catalyst-addition temperature. Polymer properties, such as gelation time, hardness, and proportional-limit elongation, are more sensitive to changes in processing than other polymer properties, such as tensile strength and polymer-to-metal bond strength. Statistical analysis of variance permitted the effects of two-factor interactions to be estimated.

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